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(54) Title: IMPROVED GLYCOL PURIFICATION

(57) Abstract

Disclosed herein are methods for producing organic liquids having superior optical properties with respect to ultraviolet light transmittance, without an attendant increase in the pH of the liquids so treated. The methods are particularly useful in producing glycols useful in polyester manufacture.

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IMPROVED GLYCOL PURIFICATION

This application claims the benefit of U. S. Provisional Application number 60/085,754 which was filed on May 14, 1998 and is currently still pending. The entire contents of said Provisional Application are herein incorporated by reference thereto.

This invention relates to the purification of organic liquids, and especially glycols, including without limitation glycols having fewer than 5 carbon atoms per molecule.

It is known that elemental carbon in various forms is suitable for use in contact treatment for removing impurities from a variety of fluid materials including air, and liquids such as water and organic liquids. Elemental carbon is known to exist in a variety of forms, including amorphous forms including soots, carbon black, charcoals, and lampblack . The macroscopic properties of these materials depend upon their particle size and surface area. Of these several forms, the charcoals, and particularly activated charcoal, find frequent employment in the treatment of the aforesaid substances. Activated charcoal is a porous, microcrystalline carbonaceous material having a surface area between about 500 to 1500 square meters of surface area per gram. It is presumed that the purification of various substances contacted with charcoals occurs through the mechanism of adsorption of various chemical species upon the surface of the charcoal.

Organic liquids including but not limited to glycols, and in particular ethylene glycol, often contain undesirable impurities acquired either through the manufacturing process from which they were spawned or from packaging and conveyance. It is towards the reduction or complete removal of impurities from organic liquids, including ethylene glycol, to which the present invention is directed.

One physical property useful for measuring the quantity of certain undesirable impurities in ethylene glycol is the percent transmittance of ultraviolet light ("uv") having

wavelengths in the 220-350 nanometer range. It is the knowledge of the inventors hereof that treatment of ethylene glycol by contacting it with various commercially available activated carbons, charcoals, and the like is capable of increasing the uv transmittance at the aforementioned wavelengths. However, an undesirable consequence of such contacting treatment is that the pH level of the glycol treated is typically caused to increase from a bout 6.5 to 7.5 to about 8.0 to 10.0, depending upon the type and grade of carbon used. This pH-raising effect of activated charcoal is easily demonstrated. For example. when fifty (50) grams of Coconut carbon (TIGG 1230) is added to 150 grams of purified ethylene glycol having a pH of 7.0 and the mixture is sealed from the air and permitted to stand with only occasional stirring, after two days the pH of the glycol (following routine filtration) is observed to be 9.1. While treated glycols having pH levels in these upper ranges are suitable for some end uses, other applications require a glycol having a more neutral pH, such as glycols intended for use in polyester production.

It is also known in the art that the use of pre-treated carbons, such as acid-washed carbon, can minimize the pH-altering effect observed when treating glycols with carbons. However. for reasons unknown, glycols treated with such carbons display poorer uv light transmittance after such treatment than before. It is indeed unfortunate that some unknown residual species tends to have an adverse effect on the uv transmittance of the glycols treated.

Thus, it has been found impossible to date to provide a treatment for increasing the uv transmittance of glycols which does not cause an attendant increase in the pH level of the glycol treated.

However, the inventors hereof have now unexpectedly discovered a process for treatment of various commercial charcoals which provides for an increased uv light transmittance with no attendant significant increase in pH of various liquids (including

but not limited to glycols) contacted with charcoals treated according to the teachings herein set forth.

SUMMARY OF THE INVENTION

This invention relates to a process for treating any one of a variety of organic liquid s to impart superior ultraviolet light transmittance characteristics to the organic liquid treated in accordance herewith, without causing an attendant increase in the pH of the liquid which comprises the steps of: providing a charcoal, contacting the charcoal with an inorganic acid, rinsing the charcoal with a solvent, optionally drying the charcoal, contacting the organic liquid to be treated with the charcoal so treated, and collecting the organic liquid. Organic liquids so treated, and especially glycols so treated when the inorganic acid is a phosphoric acid, are typically observed to possess a pH of 8.2 or less and a uv light transmittance at a wavelength of 220 nanometers (nm) of at least 70 %. This represents a substantial advantage over prior art methods, which in the case of glycols is especially beneficial when the glycol is to be employed as a raw material in the manufacture of polyester materials, according to methods known to those skilled in the art of polyester manufacture.

DETAILED DESCRIPTION

A volume of data have been generated to support the unexpected discoveries disclosed herein, including pH values and % transmittances for uv light. A Perkin-Elmer Lambda 9 UV/VIS Spectrophotometer was employed for the uv transmittance values herein set forth, and the pH values were determined using a Metrohm 744 pH meter.

As is evident from reading the Tables which follow, various carbons were treated for purposes of obtaining the data herein set forth. The carbons Norit®, and Darco® were obtained from the Aldrich Company 1001 West Saint Paul Avenue, Milwaukee, WI

53233. The Sorbonorit carbon is available from Norit in Amersfoort, the Netherlands. The Calgon carbons are available from Calgon Corporation of Pittsburgh, Pennsylvania.

Table I below illustrates that various substances are capable of affecting the uv light transmittance of ethylene glycol.

Example No.	Adsorbent	UV scan for % Transmittance, nm			
		350	275	250	220
1	None	96.7	59.1	45.3	53.2
2	Mole Sieve 5A	93.8	49.1	44.3	33.2
3	Mole Sieve 4A	67.7	20.6	16.2	8.2
4	Mole Sieve 13X	71.5	29.8	21.7	9.6
5	Clay-24 (Granular)	87.1	46.0	34.3	28.5
6	Clay-13 (Powder)	58.7	13.7	7.8	1.4
7	Activated C (Norit)	94.2	92.4	89.1	58.1

Table I

The results set forth in Table I were obtained by combining 5.0 grams of the listed adsorbent with 100 grams of impure ethylene glycol using gentle stirring at room temperature for two days. The glycol was then filtered to remove the solid carbon and the uv transmittance was subsequently determined at the wavelengths listed. By far the material having the most effect on the uv light transmittance of the glycol is the activated carbon known as Norit®.

The fact that a wide variety of carbons may be used for improving the uv light transmittance of glycols is evidenced by the data in Table II below, which was generated by filling a standard chromatography column with a 100 milliliter volume of each of the carbons set forth in Examples 8 through 54 of Table II, and trickling ethylene glycol

slowly through the column and subsequently collecting the samples. The column labeled "mL col." refers to the total number of milliliters of ethylene glycol collected (the amount which had been passed through the column) at the time the measurements were made. A value of zero mL col. represents the results for a blank, untreated sample.

Example No.	Carbon	mL col.	UV, % Transmittance at (nm)			
			350	275	250	220
8		0	93.5	56.8	43.2	50.7
9	Aldrich (Norit)	200	96.9	93.6	89.8	65.2
	RO 0.8					
10		400	98.2	95.7	92.7	77.7
11		600	98.5	95.9	93.1	81.0
12		800	98.2	94.9	91.8	78.3
13		0	92.3	55.4	41.9	48.7
14	Calgon Act. C	200	93.5	87.6	93.2	60.6
	F-300 (8x30)					
15		400	99.0	95.8	92.7	77.9
16		600	98.0	93.9	90.2	75.8
17		800	95.5	88.2	83.8	69.0
18		1000	100.0	95.3	91.8	79.9
19		0	98.4	69.4	59.2	58
20	Calgon Act. C	200	95.3	83.3	77.9	51.0
	SRBMW (4x10)					
21		400	97.9	85.8	80.3	65.7
22		600	98.8	87.1	81.8	70.5
23		800	99.7	96.8	81.2	69.9
24		1000	99.5	86.4	80.7	70.5
25		0	96.7	67.9	57.8	55.9
26	Darco Act. C	200	99.8	91.9	86.9	50.2

	18-89 (4x10)					
27		400	100.0	94.9	91.0	75.1
28		600	99.7	93.5	89.3	72.1
29		800	100.0	94.6	90.4	75.6
30		1000	100.0	93.3	88.6	73.1
31		0	97.8	69.4	59.2	58.3
32	Sorbonorit B3	200	98.8	86.8	80.8	65.9
33		400	100.0	89.3	84.0	72.0
34		600	100.0	88.0	82.2	70.5
35		800	99.4	83.0	75.9	68.7
36		1000	99.7	80.9	73.0	66.8
37			97.9	69.1	58.9	57.1
38	Calgon Act. C	200	96.8	83.3	77.1	65.6
	CPN(IV) (4x6)					
39		400	99.7	83.7	77.0	69.2
40		600	99.2	81.7	74.3	66.9
41		800	98.8	80.8	73.3	66.6
42		1000	99.6	81.3	73.7	67.3
43		0	98.5	69.5	59.5	57.3
44	Calgon Act. C	200	98.4	86.3	80.5	66.4
	BPL (4x10)					
45		400	99.7	89.1	84.0	72.9
46		600	99.7	87.6	81.9	71.8
47		800	99.1	86.0	80.0	70.0
48		1000	98.9	85.6	79.3	69.3
49			98.0	69.4	59.2	57.8
50	Columbia C	200	96.8	83.5	77.2	63.9
	933-03					
51		400	99.7	87.3	81.2	70.9

52			600	99.3	85.9	79.3	67.9
53			800	93.6	79.1	72.1	60.9
54			1000	94.5	78.5	70.6	57.8

Table II

A 245 milliliter volume of the activated carbons shown in Examples 55 through 105 in Table III. below were charged to a 1 " x 30" stainless steel tubular reactor and the carbons were permitted to be in contact with ethylene glycol for a time sufficient to displace all air from the pores of the carbons. The ethylene glycol was then run through the carbon beds at the rate indicated. Samples were taken after the times indicated and the UV % transmittance determined.

Example No.	Activated Carbon	Time	T	React. P	F. R.		Transmittance, %, at nm		
		(hrs)	deg. C	(Psig)	(ml/min)	350	275	250	220
55		0				96.8	81.3	73.3	61.2
56	TIGG AWC-	1.0	65	0	10	78.4	54.4	25.6	1.2
57	1240	3.0	68	0	10	89.6	73.3	48.9	8.9
58		4.0	70	0	10	92.6	79.7	59.1	16.5
59		5.0	70	0	10	92.9	82.7	70.3	36.0
60		6.0	70	0	10	88.6	79.0	64.4	23.8
61		8.0	69	0	10	73.5	64.0	53.7	24.4
62		9.0	71	0	10	84.3	75.3	66.1	35.9
63		1.0	25	0	10	40.9	31.8	21.6	6.0
64		2.5	25	0	10	75.1	65.1	59.8	38.0
65		3.5	26	0	10	98.0	94.9	90.7	68.3
66		4.5	24	0	10	98.9	96.7	93.3	70.4
67		0.0				97.9	83.7	77.0	66.6
68	TIGG Type	0.5	60	0	10	83.6	86.0	68.2	31.8
69	CC 1230	1.5	70	0	10	97.3	95.2	91.5	73.1
70	Coconut	2.5	63	0	10	98.4	96.2	93.1	73.7
71	Carbon	4.5	64	0	10	98.2	95.6	92.9	74.1
72		6.0	64	0	10	98.9	96.9	94.8	78.9
73		1.0	23	0	10	97.3	96.2	93.9	77.6
74		2.7	23	0	10	100.9	100.2	98.1	80.0
75		3.5	24	0	10	99.0	97.1	94.7	77.9

76		6.0	24	0	10	98.2	97.0	95.1	80.3
77		0.0				100.7	86.9	80.0	69.1
78	Norit Act. C	1.0	71	0	5	100.6	96.9	91.6	56.9
79	RO 0.8	2.0	70	0	5	99.6	96.6	92.2	63.9
80	pellets	3.5	72	0	5	99.2	95.1	90.5	63.0
81		4.5	71	0	5	98.7	93.7	88.9	61.0
82		6.0	70	0	5	100.0	95.7	91.6	66.7
83		1.0	22	0	4.5	99.9	97.8	95.2	77.9
84		2.5	21	0	4.5	99.9	97.6	94.8	78.1
85		3.5	22	0	4.5	100.7	98.5	96.1	81.8
86		5.5	22	0	4.5	99.5	97.0	94.4	77.5
87		7.0	22	0	4.5	100.0	97.4	94.4	75.4
88		0.0				99.6	85.3	77.5	64.5
89	TIGG Type	0.8	27	100	7.5	96.2	90.0	73.6	21.3
90	CC 1230	2.0	23	100	9.3	98.4	93.5	85.1	38.5
91	Coconut C	3.2	24	100	10.2	99.3	95.1	88.8	48.1
92		4.3	25	100	9.7	99.2	95.4	90.7	54.9
93		5.0	26	100	10.0	99.8	96.0	91.5	57.9
94		6.7	27	100	9.3	99.4	95.4	91.1	61.2
95		7.8	27	100	9.3	100.0	96.4	92.1	62.1
96		8.8	29	100	9.3	94.5	96.4	82.5	56.1
97		10.0	23	100	10.2	99.1	96.2	93.7	70.1
98		12.2	23	100	10.5	100.0	98.4	95.7	76.0
99		14.3	25	100	9.7	100.0	97.7	94.8	75.6
100		16.7	26	100	9.7	100.0	97.2	94.2	74.8
101		17.7	22	100	10.7	100.0	98.6	95.4	72.4
102		20.0	24	100	10.7	100.0	96.7	93.4	76.4
103		22.1	24	100	10.5	100.0	98.5	95.3	78.6
104		24.2	24	100	10.5	100.0	97.9	95.6	83.0
105		25.4	24	100	9.0	100.0	96.8	93.7	76.7

Table III

The first carbon in Table III. (TIGG AWC 1240) is an acid washed Coconut carbon as received from TIGG Corporation. The poor uv light transmittance quality of glycol treated with this material becomes evident when compared with all of the other carbons in this table. none of which had been acid washed.

To treat the various commercial charcoals for rendering a carbon suitable for use in accordance with the organic liquid (esp. glycols) treatment process of this invention. one first provides the carbon to be treated. This can be any charcoal material regardless of the material from which it is derived. It is preferred. however. that the charcoal is derived from coconut shells. Then. phosphoric acid dissolved in a suitable carrier. such

as an alcohol, is contacted with the carbon for a time period sufficient to inhibit subsequent alkalinization of glycol contacted with the carbon. As an example, to a column measuring 1" in diameter and 30 " long is charged a 250 ml volume of fresh charcoal derived from coconut shells, specifically Coconut Carbon "TIGG 12X30" from Tigg Corporation of 800 Old Pond Road, Suite # 706, Bridgeville, Pennsylvania 15017. In a flask are admixed 1.2 grams of phosphoric acid and one liter of pure methanol. The acidified methanol is then run through the carbon-charged column at a rate of 10 milliliters per minute, while the temperature is maintained at between about 25 and 30 degrees Centigrade. Next, the column is flushed using pure methanol, flowing through the column at a rate of 10 milliliters per minute for one hour. Next, the temperature of the column is increased to 50 degrees centigrade over the course of 15 minutes, and the flow of pure methanol is continued for an additional two hours. The methanol flow is discontinued, and a purge of Nitrogen gas at 1 Liter per minute through the column is commenced while the temperature of the column is raised to 100 degrees centigrade over the course of one hour. The temperature of the column is then maintained at 100 degrees centigrade for 5 hours with the Nitrogen purge remaining on all the while. Finally, the column and its contents are permitted to cool to room temperature by the removal of the heat source. Next, the reactor column was contacted with ethylene glycol for a time sufficient to remove all air bubbles from the pores of the carbon. Ethylene glycol was pumped through the reactor column at 100 psig reactor pressure and the temperature and flow rate indicated in Examples 106 through 133 in Table IV. below:

Example No.	Time	T	F. R.	Transmittance,	at	nm		pH
	(hrs)	(deg C)	(ml/min)	350	275	250	220	
106	0			99.8	84.5	76.7	63.9	6.8
107	2	22	11.8	99.5	97.4	94.0	71.9	8.2
108	4	22	9.8	100.0	96.9	93.2	71.3	8.5
109	6	22	10.6	99.2	96.1	93.1	75.7	8.7
110	8	24	10.6	99.8	96.5	93.8	78.5	8.8
111	10	21	10.4	99.7	97.1	95.3	83.8	8.7
112	12	21	10.4	100.0	96.4	92.9	74.0	8.8
113	14	22	10.2	100.0	96.9	93.4	75.5	8.7
114	16	23	10.3	100.0	97.0	93.7	77.8	8.6
115	18	22	10.5	100.0	98.5	95.6	78.4	8.5
116	20	23	10.3	100.0	97.6	94.1	77.0	8.7
117	22	23	10.1	100.0	96.0	92.3	75.8	8.7
118	24	24	10.1	100.0	96.9	93.3	78.0	8.5
119	26	24	10.3	100.0	99.4	97.4	85.2	8.6
120	28	25	10.3	100.0	97.6	95.0	.83.0	8.4
121	30	25	10.4	100.0	96.6	93.5	78.7	8.5
122	32	25	10.2	99.0	94.4	90.8	76.5	9.6
123	34	23	10.2	100.0	97.7	95.4	83.5	8.6
124	36	24	10.3	100.0	97.1	94.3	81.8	8.2
125	38	25	10.3	99.6	94.8	91.4	77.4	8.2
126	40	25	10.3	99.8	94.7	91.3	78.9	8.1
127	42	23	10.6	100.0	96.6	93.2	78.5	8.4
128	44	25	10.3	99.8	95.4	92.1	79.6	8.1
129	46	25	10.0	100.0	96.0	92.4	78.9	8.0
130	48	24	9.3	99.6	96.5	93.7	82.6	7.9
131	50	25	9.1	100.0	97.1	93.9	81.8	7.9
132	52	25	10.6	100.0	96.3	92.6	80.5	
133	54	25	10.6	100.0	96.9	93.1	80.3	8.5

Table IV

Conditions identical to those employed to generate the data set forth in Examples 106 through 133 in Table IV. above were employed for generation of the data listed in Table V below, excepting that the amount of Phosphoric acid used was 12.0 grams per liter instead of 1.2 grams. Again, the reactor column was contacted with ethylene glycol for a time sufficient to remove all air bubbles from the pores of the carbon. Ethylene glycol was pumped through the reactor at 100 psig reactor pressure and the temperature and flow rate indicated in Examples 134 through 173 in Table V. below:

Example N .	Tim	T	F. R.	Transmittance,	at	nm		pH
	(hrs)	(Deg C)	(ml/min)	350	275	250	220	
				99	95	92	70	
134	0			92.2	68.4	61.4	43.9	6.5
135	2	24	9.8	99.5	97.6	95.3	80.8	5.9
136	4	26	9.8	100.0	98.6	96.5	84.4	
137	6	27	10.2	100.0	98.4	95.8	82.2	6.4
138	8	58	10.2	100.0	96.4	92.7	77.0	
139	10	52	10.2	99.7	96.8	93.5	76.6	7.4
140	12	46	10.3	98.3	94.5	90.4	76.6	
141	14	54	10.4	100.0	98.2	95.3	81.0	6.6
142	16	46	10.2	100.0	98.4	95.3	79.6	
143	18	51	10.2	100.0	97.6	94.4	80.6	6.6
144	20	51	10.0	100.0	96.8	93.4	80.3	
145	22	48	10.3	99.8	97.5	94.8	82.6	
146	24	53	10.3	95.7	91.3	87.0	67.7	
147	26	72	9.8	99.5	95.7	91.4	75.3	
148	28	55	9.8	100.0	96.3	92.5	78.2	
149	30	23	10.3	100.0	98.3	95.7	82.5	6.2
150	32	23	10.3	100.0	98.0	95.2	82.1	
151	34	23	10.3	100.0	98.4	95.3	79.5	
152	36	24	9.8	99.2	96.1	92.5	77.0	7.4
153	38	25	9.8	100.0	97.1	93.6	78.5	
154	40	24	9.8	95.5	92.0	88.9	77.8	
155	42	24	9.8	99.0	95.6	93.3	75.6	7.1
156	45	24	9.8	100.0	98.1	95.1	81.6	
157	47	24	9.9	100.0	98.2	95.6	84.1	
158	49	25	10.0	100.0	96.1	93.1	82.4	7.1
159	51	25	9.9	100.0	97.3	94.5	85.2	
160	53	25	9.9	100.0	96.1	91.8	79.4	
161	55	23	10	100.0	97.0	94.0	80.9	7.0
162	57	23	9.5	99.9	95.8	92.3	80.7	
163	59	24	10.5	100.0	95.8	92.2	80.2	6.8
164	61	24	10.5	99.3	94.1	90.1	78.9	
165	63	23	10.0	100.0	97.4	93.8	80.0	7.6
166	65	23	10.0	100.0	96.2	92.1	78.5	
167	67	23	9.9	100.0	95.7	91.5	78.9	7.7
168	69	24	9.9	100.0	95.7	91.4	78.9	
169	71	23	10.1	100.0	96.7	92.8	78.8	
170	73	23	10.0	100.0	95.5	91.3	79.4	7.7
171	75	24	10.0	100.0	95.3	90.6	77.4	
172	77	23	10.0	100.0	96.2	92.2	79.6	
173	79	23	10.0	99.9	94.4	90.2	78.4	7.7

Table V

In order to validate the unexpected results obtained from the treatment regiments employed herein as illustrated in examples 134 - 173, a 250 milliliter volume of TIGG (12x30) activated carbon was charged to the reactor column employed above in Examples

55 through 173. The contents of the column were then contacted with ethylene glycol to remove all air bubbles from the pores of the carbon. Ethylene glycol was pumped through the reactor at 100 psig reactor pressure and the temperature and flow rate indicated in the table below.

Example No.	Time	T	F.R.	Transmittance, % at nm,				pH
	(hrs)	deg C	(ml/min)	350	275	250	220	
174	0			99.8	84.5	76.7	63.9	6.8
175	2	48	9.8	99.6	98.3	94.6	76.1	
176	4	45	9.7	100.0	97.2	93.9	74.1	
177	6	48	10.3	98.6	96.2	92.7	78.1	9.9
178	8	46	9.9	100.0	98.1	96.7	82.3	
179	10	45	10.3	100.0	99.6	97.8	82.9	
180	12	50	10.3	100.0	99.5	97.8	82.6	9.5
181	14	49	11.0	100.0	98.9	97.5	84.9	
182	16	50	11.0	100.0	98.3	96.8	84.6	
183	18	50	10.2	100.0	97.6	95.5	80.3	9.4
184	20	50	10.2	100.0	96.7	94.5	79.0	
185	22	39	10.5	100.0	97.9	95.8	81.0	
186	24	48	10.5	100.0	97.5	95.1	80.7	9.2
187	26	48	9.9	100.0	97.5	94.9	79.4	
188	28	54	10.0	100.0	97.2	95.0	82.0	
189	30	50	10.2	100.0	97.3	95.3	82.9	9.1
190	32	50	10.2	100.0	96.1	93.5	80.2	
191	34	50	9.6	100.0	96.8	94.2	82.1	
192	36	44	9.9	100.0	97.9	95.2	80.4	9.0

Table VI

After consideration of the results of Examples 174 through 192 in light of earlier experimental results herein set forth. it becomes evident that high pH ethylene glycol is obtained if the carbon is not acid washed prior to treatment of the glycol therewith. However. as is also evident from the data concerning commercially-available acid washed carbons. (as seen in Examples 55-62), the commercially available acid-washed carbons tend to give poor performance when it comes to uv light transmittance characteristics. Examples 106 through 173 show that for the first time, by the instant invention, it is now possible to provide ethylene glycol having a high uv light transmittance and at a relatively neutral pH

level, thus making glycols treated by the process herein especially well suited for use in preparing polyesters.

For purposes of this invention, any activated carbon may be utilized as the media to which the organic liquid is exposed in order to improve its uv light transmittance characteristics while simultaneously not increasing the pH level of the organic liquid any appreciable extent. However, owing to economic considerations, charcoal is the generally preferred amorphous form of carbon for use in accordance hereto, and charcoal derived from coconut shells is especially preferred.

The carbons used in accordance hereto are contacted with an inorganic acid prior to being contacted with the organic liquid. For this purpose, any inorganic acid, or its solutions in water or organic solvents may be used. Suitable organic solvents include all those solvents in which the acid used is miscible. Preferred solvents may be selected from the group of: polar organic solvents including: alcohols, ethers, and glycols.

Subsequent to being contacted with an inorganic acid, the carbon employed may be rinsed with either water or an organic solvent selected from those mentioned above as being suitable solvents for the inorganic acid. The same solvent used to dissolve the inorganic acid may be used in pure form as the rinse agent.

Following the rinse step, the carbon may, although not necessarily, be dried. Drying is beneficial in cases when the rinse solvent is different from the organic liquid to be treated. It is preferred, however, to rinse the carbon prior to its being contacted with the liquid which is to be treated with some of the liquid to be treated. That is, when ethylene glycol is to be purified in accordance with the invention, it is beneficial to use purified ethylene glycol as the rinse agent, in order to avoid the necessity of drying. The carbon may be dried as is convenient to reduce its weight, as in cases where the carbon is treated at a physical location different from where the organic liquid is to be treated, in order to reduce freight costs by

lowering the weight of the carbon. During the drying step, if elected, it is beneficial to increase the temperature of the column in which the carbon is preferably contained to a temperature in the range of about 0 to 200 degrees centigrade, more preferably between 20 and 150 degrees centigrade, and most preferably between 50 degrees centigrade and 120 degrees centigrade, with 100 degrees centigrade being most preferable. The drying is carried out by supplying one of the two ends of said column in which the carbon is contained with a source of pressurized gas. For this purpose, any gas which does not react with nor is adsorbed onto the carbon to any appreciable extent may be employed for this purpose. For such use nitrogen, hydrogen, or any of the noble gases may be employed, with nitrogen being most preferred owing to its relatively low cost and widespread availability.

Following the rinsing (or the drying step when opted) the carbon that is preferably contained in a column, is permitted to come into thermal equilibrium with the ambient surroundings and is contacted with the organic liquid to be treated. Initially, there may be air bubbles present, and the column is purged with the liquid to be treated until air bubbles are no longer seen emerging from the exit portion of the tube, at which time the aliquot which was collected while bubbles were emerging may be discarded and the collection of the desired product from the exit end of the column commenced. Preferably, the organic liquid to be treated is pumped through at a rate of between 10 and 500 psig per square inch of diameter in which the carbon is contained, with 128 psig per square inch of column cross sectional area being most preferred. Preferably, the column is constructed of a material which is inert to the organic liquid to be treated and is strong enough to maintain the pressures encountered. For this purpose, a stainless steel column is preferred. The column length may be any length sufficient to produce an organic liquid possessive of the properties desired. However, columns of at least 20 inches in length are generally required, with 30 inches being most preferable.

Consideration must be given to the fact that although this invention has been shown and described with respect to certain preferred embodiments. it is obvious that equivalent alterations and modifications will occur to others skilled in the art upon the reading and understanding of the specification, such as improving the optical qualities of any liquid material known in the chemical arts while not causing an attendant increase in the pH of such liquid. The present invention includes all such equivalent alterations and modifications, and is limited only by the scope of the claims which follow.

We claim:

1) A process for treating an organic liquid to impart superior ultraviolet light transmittance characteristics to the organic liquid without causing a substantial increase in the pH of the liquid which comprises the steps of:

a) providing a charcoal;

b) contacting said charcoal with an inorganic acid;

c) rinsing said charcoal with a solvent;

d) optionally drying said charcoal;

e) contacting said organic liquid with said charcoal; and

f) collecting the organic liquid,

wherein said organic liquid collected has a pH of 8.2 or less and a uv light transmittance at a wavelength of 220 nanometers (nm) of at least 70 %.

2) A process according to claim 1 wherein said inorganic acid is selected from the group consisting of: perchloric acid, chloric acid, chlorous acid, hypochlorous acid, bromic acid. bromous acid, hypobromous acid, periodic acid, iodic acid, iodous acid, and hypoiodous acid.

3) A process according to claim 1 wherein said inorganic acid is selected from the group consisting of: sulfuric acid, sulfurous acid, hydrochloric acid, hydrofluoric acid, hydrobromic acid, hydriodic acid, phosphoric acid, phosphorous acid, fluosilicic acid, nitrous acid, and nitric acid.

4) A process according to claim 3 wherein said inorganic acid is a solution of inorganic acid in a solvent selected from the group consisting of: water, alcohols, ethers, and glycols.

5) A process according to claim 4 wherein the concentration of inorganic acid in said solvent is between about 0.01 % and 85 % by weight, including every hundredth percentage therebetween.

6) A process according to claim 5 wherein the concentration of inorganic acid in said solvent is between about 1 % and 15 % by weight.

7) A process according to claim 1 wherein said organic liquid is selected from the group consisting of : alcohols, ethers, glycols, polyalkylene glycols, and alkylene carbonates.

8) A process as in claim 7 wherein said organic liquid is a glycol is selected from the group consisting of: ethylene glycol, propylene glycol, and butylene glycol.

9) A process according to claim 7 wherein said organic liquid is a polyalkylene glycol having alkylene portions containing between 2 and 5 carbon atoms per alkylene moiety.

10) A process according to claim 1 wherein said solvent comprises water.

11) A process according to claim 1 wherein said solvent comprises an organic solvent.

12) A process according to claim 11 wherein said organic solvent comprises a solvent selected from the group consisting of: alcohols, glycols, and ethers.

13) A process according to claim 1 wherein said solvent comprises a solvent selected from the group consisting of: methanol, ethanol, propanol, butanol, and isopropanol.

14) A process according to claim 5 wherein the temperature at which the charcoal is maintained during its contact with said inorganic acid is in the range of from about the freezing point of the inorganic acid solution to 100 degrees centigrade, including every whole integer degree centigrade therebetween.

15) A process according to claim 1 wherein the temperature at which the charcoal is maintained during the rinsing step is between about 0 and 100 degrees centigrade, including every whole integer degree centigrade therebetween.

16) A process according to claim 1 wherein the drying step is carried out by contacting the charcoal with a gas, said drying being conducted at a temperature in the range of about 0 degrees centigrade and 200 degrees centigrade, including every whole integer degree centigrade therebetween.

17) A process according to claim 14 wherein said gas includes at least one gas selected from the group consisting of: nitrogen, hydrogen, and the noble gases.

18) A process according to any of claims 1 through 17 wherein said drying is carried out under reduced pressure.

19) A process according to any of claims 1 through 18 wherein the organic liquid is contacted with said charcoal for an effective amount of time to cause the organic liquid collected to have a uv light transmittance at 250 nm of at least 90 % and a pH less than 8.2.

20) A process according to any of claims 1 through 19 wherein the organic liquid is contacted with said charcoal for an effective amount of time to cause the organic liquid collected to have a uv light transmittance at 275 nm of at least 90 % and a pH less than 8.2.

21) A process according to any of claims 1 through 20 wherein the organic liquid is contacted with said charcoal for an effective amount of time to cause the organic liquid collected to have a uv light transmittance at 350 nm of at least 90 % and a pH less than 8.2.

22) A process according to any of claims 1 – 21 wherein said inorganic acid contains at least one acid selected from the group consisting of: ortho phosphoric acid, pyrophosphoric acid, or polyphosphoric acid.

23) An organic liquid collected from a process according to any of claims 1 – 22.

24) A polyester product produced from a glycol which is collected from a process according to any of claims 1 –23.

INTERNATIONAL SEARCH REPORT

Intr   ional Application No
PCT/US 99/10247

A. CLASSIFICATION F SUBJECT MATTER
IPC 6   C07C29/76

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6   C07C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 5 294 305 A (F.S. CRAFT, SR., ET AL.) 15 March 1994 (1994-03-15) column 2, line 30 - line 63; claim 20	1
A	US 3 970 711 A (C.R. REICHE, ET AL.) 20 July 1976 (1976-07-20) claim 1; example	1

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

° Special categories of cited documents :

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Date of the actual completion of the international search	Date of mailing of the international search report
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INTERNATIONAL SEARCH REPORT

Information on patent family members

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PCT/US 99/10247

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